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ROT-AND WEATHER-RESISTANCE OF METHYLOLMELAMINE-TREATED COTTON FABRICS

by

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and

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December 1971

UNITED STATES ARMY
NATICK LABORATORIES
NATICK, MASSACHUSETTS 01760

Pioneering Research Laboratory

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13. ABSTRACT

White cotton fabrics treated to contain 9 to 12% methylolmelamine resin, which should be sufficient for optimum long-term environmental protection, were subjected to extended soil burial and outdoor weathering exposure. The resin was applied either by the "dry-cure" process of Berard or by one of two "wet-cure" processes – the "Arigal" process or the "Statuff" process. Dyed Army fabrics were included in the tests to determine the effectiveness of "Arigal" treatment in a potential end-item such as lightweight jungle uniforms and the efficacy of a lower add-on level.

Fabrics treated to contain 9 to 12% methylolmelamine resin by the "Arigal" process and the "dry-cure" process of Berard were quite resistent to microbiological degradation during extended soil burial. The fabric prepared by the "Statuff" process, which was not as resistant, was actually less rot-resistant than "Arigal" treated fabrics containing only 7 to 8% resin. The latter fabrics were not severely affected by 4 months of soil burial exposure performed under our screening program. Despite differences between processes, all treated fabrics containing 9 to 12% resin weathered excellently outdoors. After outdoor exposure fabrics treated either by the "dry-cure" process of Berard or the "Arigal" process and originally containing 9 to 12% resin were still substantially rot-resistant – considerably more so than fabric prepared by the "Statuff" process.

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FOREWORD

This report is an evaluation of the long-term rot- and weather-resistance of cotton fabrics treated with methylolmelamine resins. The study was conducted to compare the protection afforded by available "wet" and "dry-cure" methods of applying methylolmelamine.

We acknowledge the efforts of Dr. A. G. Kempton who was in the NLABS Applied Microbiology Group at the inception of this study and contributed significantly to the success of this undertaking. Mr. W. N. Berard of the Department of Agriculture contributed technical expertise throughout the study and assisted in the procurement of test fabric treated by his dry-cure process.

Mr. Harry T. Skerritt of the NLABS C&PLSE Lab performed the laundering tests and supplied Table II. Mr. Carlo G. De Marco, formerly in the NLABS Functional Finishes and Leather Branch, coordinated some of the effort between industry and the NLABS Applied Microbiology Group.

The climatological data tabulated in the Appendix were gathered and compiled by the U.S. Army Meteorological Team stationed at the NLABS Sudbury Annex.

The work was accomplished under project number 1J062110A031-01.

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ABSTRACT

White cotton fabrics treated to contain 9 to 12% methylolmelamine resin, which should be sufficient for optimum long-term environmental protection, were subjected to extended soil burial and outdoor weathering exposure. The resin was applied either by the "dry-cure" process of Berard or by one of two "wet-cure" processes – the "Arigal" process or the "Statuff" process. Dyed Army fabrics were included in the tests to determine the effectiveness of "Arigal" treatment in a potential end-item such as lightweight jungle uniforms and the efficacy of a lower add-on level.

Fabrics treated to contain 9 to 12% methylolmelamine resin by the "Arigal" process and the "dry-cure" process of Berard were quite resistant to microbiological degradation during extended soil burial. The fabric prepared by the "Statuff" process, which was not as resistant, was actually less rot-resistant than "Arigal" treated fabrics containing only 7 to 8% resin. The latter fabrics were not severely affected by 4 months of soil burial exposure performed under our screening program. Despite differences between processes, all treated fabrics containing 9 to 12% resin weathered excellently outdoors. After outdoor exposure fabrics treated either by the "dry-cure" process of Berard or the "Arigal" process and originally containing 9 to 12% resin were still substantially rot-resistant – considerably more so than fabric prepared by the "Statuff" process.

Rot- and Weather-Resistance of Methylolmelamine-Treated Cotton Fabrics

1. Introduction

There has been considerable interest in the use of resin finishes to render cotton weather- and rot-resistant. Cotton fabric when treated with methylolmelamine resin can achieve a degree of protection unmatched by conventional add-on fungicides. Some observers attributed the microbiological resistance to a physical barrier of insolubilized resin. This view is supported by data from enzymatic studies performed on cellulose (7). However, there is direct chemical evidence for covalent bonding between the methylolmelamine and the cellulose regardless of whether the fabric is cured in the "wet" or "dry state" (16, 17). It is quite likely that the resistance and permanence of methylolmelamine finishes are dependent on the physico-chemical nature of the "resin-cellulose complex".

The rot-resistance of cotton treated with condensation resins by wet-fixation was described by Ruperti in 1956 (11). Fungal resistance was effected with minimal detriment to the natural mechanical properties of the fabric. The treatment as originally proposed was inconvenient for textile finishing plants. Specialized equipment and lengthy batch processing were required. However, in 1961 Ruperti demonstrated that wet-fixation could be performed in a continuous steam ager (12). Subsequently, wet-fixation became the basis for two competitive commercial finishes – the "Arigal" process involving the use of a powdered methylolmelamine resin with hydrogen peroxide as the acid former and the "Statuff" process employing a liquid methylated methylolmelamine and acid-forming metal salts. Either finish could be applied by batch wet-ageing at room temperature or steaming at elevated temperatures.

The evaluation of cotton fabrics treated in the wet-state complements a previous paper from this laboratory concerned with fabric treated by the formic acid colloid process -- a "dry-cure" technique (6). This fabric had been treated according to the recommendations of Berard, *et al*, for achieving maximum rot-resistance. i.e., freshly prepared colloidal solution containing 20% formic acid and sufficient methylolmelamine to yield a 12% resin add-on (1). The solution was applied by the conventional pad, dry and cure technique common to resin finishes. Under environmental stress the treated fabric lost no breaking strength during 64 weeks of soil burial and only 1/3 of the breaking strength after 21 months' outdoor exposure. The control lost 1/3 after 2 months outdoors. In effect, the finish had outperformed Berard's claims. A cause for serious objection is the excessive breaking strength loss caused by Berard's process but which does not occur when fabric is treated by wet-fixation methods. However, Rogers and Kaplan demonstrated that the breaking strength could be improved by autoclaving (10). Boyle showed that the breaking strength loss common to "dry-cure" finishes could be minimized by the addition of a strong hydrogen bonding agent to the resin bath (2). Boyle's studies are the basis for a later "Statuff" finish which should not be identified with the earlier wet-fixation process.

This report supplements a previous publication from this laboratory (5) and contains completed data obtained from the extended soil burial and outdoor weathering of fabrics treated in the "dry-state" by Berard's process and in the "wet-state" by the "Arigal" and "Statuff" processes. Physical and chemical measurements were employed to differentiate between and determine the extent of actinic or microbiological damage. Comparisons are drawn between fabrics treated by "wet-" and "dry-cure" methods through use of the chemical and breaking strength data contained in this report and other results previously published.

2. Materials and Methods

a. Fabrics

The fabrics evaluated in this study are listed in Table I. Treatment in the "dry-state" was performed with freshly prepared colloidal trimethylolmelamine in a solution of 20% formic acid. Treatment in the "wet-state" was performed with powdered methylolmelamine and hydrogen peroxide catalyst for the "Arigal" finishes and with a liquid methylated methylolmelamine resin and acid-forming metal salts for the "Statuff" finish. All finishes were applied under the supervision of technical personnel responsible for the particular treatment. With the exception of fabrics 8, 9, 10, and 11, all finishes were applied in commercial plants under production conditions.

Comparison of the finishes was based largely on the long-term evaluation of white fabrics 1-6 and 7-9 which were two separate studies. In the first study, extended soil burial data were obtained from "blue-line" duck fabrics 1-3 and outdoor exposure data from gabardine fabrics 4-6. "Blue-line" duck was not used for the outdoor weathering study to eliminate the possibility that the blue dye might interfere with the course of actinic degradation. In the second study, the soil burial and outdoor weathering data were obtained from the same set of fabrics – "double-warp" duck fabrics 7-9. Fabrics 2, 3, 5, 6, 8 and 9 were treated to contain between 9 and 12% resin for maximum environmental protection. Variation in actual resin add-on that resulted did not significantly affect the test results.

Dyed Army fabrics 10-11 treated by the "Arigal" process and containing only 7 to 8% "Arigal" add-on resin were screened to evaluate efficacy of a low add-on level. They were the only fabrics not treated in a textile finishing plant.

Vat-dyed cotton poplin fabrics 12-14 were "Arigal" treated to obtain data for material that could be used in a potential end-item such as light-weight jungle uniforms. Fabrics 13 and 14 differ only in that a polyethylene softener was added to fabric 14. Fabrics 12 and 13 were subjected to extended soil burial and outdoor weathering tests.

TABLE I

Fabrics Tested

Fabric No.	Description Base Fabric	Type Finish	% Methylol-melamine[1]	Date Treatment	Place Treatment
1	Cotton (8 oz) "blue-line" Duck[2]	None	---	---	
2	Cotton (8 oz) "blue-line" Duck[2]	"Arigal"	9.9	9/62	Northern Dyeing Corp.
3	Cotton (8 oz) "blue-line" Duck[2]	"Statuff"	---	8/62	Cranston Print Works
4	Cotton (7.2 oz) bleached white Type II (gabardine) MIL-C-14373	None	---	---	
5	Cotton (7.2 oz) bleached white, Type II (gabardine) MIL-C-14373	"Arigal"	10.7	9/62	Northern Dyeing Corp.
6	Cotton (7.2 oz) bleached white, Type II (gabardine) MIL-C-14373	"Statuff"	---	8/62	Cranston Print Works
7	Cotton (9.85 oz) bleached white, Type III (duck) MIL-C-419a[3]	None	---	---	
8	Cotton (9.85 oz) bleached white, Type III (duck) MIL-C-419a[3]	"dry-cure"	12.2	11/64	NLABS
9	Cotton (9.85 oz) bleached white, Type III (duck) MIL-C-419a[3]	"Arigal"	11.2	6/65	Ciba
10	Cotton (6 oz) poplin	"Arigal"	7.7	5/62	Ciba
11	Cotton (8.8 oz) sateen	"Arigal"	7.3	5/62	Ciba
12	Cotton (6 oz) olive green poplin, Wind resistant, MIL-C-00342	None	---	---	
13	Cotton (6 oz) olive green poplin, Wind resistant, MIL-C-00342	"Arigal"	7.9	3/63	Kenyon Mills
14	Cotton (6 oz) olive green poplin, Wind Resistant, MIL-C-00342[4]	"Arigal"	---	3/63	Kenyon Mills

[1] By nitrogen analysis.

[2] A "standard" fabric described by Shapiro (13).

[3] A test fabric designed by Brysson and Markezich (3) to simplify the preparation of ravelled strips for breaking strength measurements.

[4] Polyethylene softener applied subsequent to the resin treatment.

b. Durability Tests

Outdoor weathering tests were performed at the NLABS Sudbury Annex previously designated the Maynard Annex. Large swatches of cloth were tacked on the same unshaded, open-backed, wooden racks used for the exposure of "dry-cured" fabric (6). The racks face south at an angle of 45° from the vertical, and fabrics were a minimum distance of 30 inches above the ground. After weathering, treated samples and controls were ravelled into 1 x 6 inch warp strips.

Weatherometer, leaching and soil burial testing were conducted on 1 x 6 inch ravelled warp strips in accordance with Methods 5670, 5830, and 5762, respectively, of Federal Specification CCC-T-191b (15).

Hydrolytic experiments were performed by refluxing yarns 2 x 6 inches in length with water in a 2-liter reaction flask. The initial ratio of yarns to liquid was 1:50 by weight before samples were withdrawn for nitrogen analysis.

Data describing the laundering tests performed in a military-type, single-trailer field washer of 60 pound capacity are listed in Table II Three 1/2 x 1 yd. swatches of each of the fabrics to be laundered were added to the field washer. One of each was removed after one standard cotton wash and the remaining two test pieces after five standard launderings. One of the two pieces laundered five times was then subjected to five cold water launderings.

c. Test Methods

All samples to be tested were conditioned at 70 ± 2°F and 65 ± 2% RH for at least 24 hours. Therefore, the results of chemical analyses contain a small but constant error term due to moisture content.

Breaking strengths were measured on an Instron Tensile Tester Model TT-CI and tearing strengths on an Elmendorf Tear Test Machine according to Methods 5104.1 and 5132, respectively, of Federal Specification CCC-T-191b (15).

Fluidity and solubility measurements were performed as described by Clibbens and Little (4) and Mehta and Mehta (8), respectively. Nitrogen content was determined with a Thomas-ASTM microKjeldahl apparatus.

3. Results and Discussion

a. Some Preliminary Observations

The results of physical tests which were performed prior to the long-term evaluations are listed in Table III. Neither "wet-cure" treatment caused the appreciable breaking strength loss experienced with "dry-cured" fabrics. However, tear strength losses generally

TABLE II

Laundering Tests Performed in a Military-Type, Single-Trailer Field Washer of 60 Pound Capacity

OPERATION	STANDARD COTTON WASH				COLD WATER COTTON WASH			
Description	Level In.	Time	Temp. °F	Detergent Oz.[1]	Level In.	Time	Temp. °F	Detergent Oz.[2]
Suds	8	5 Min.	100	6	8	5 Min.	60	6
Suds	8	5 Min.	130	3	8	5 Min.	60	3
Suds	8	5 Min.	130	2	8	5 Min.	60	2
Rinse	11	2 Min.	130	--	11	2 Min.	60	--
Rinse	11	2 Min.	130	--	11	2 Min.	60	--
Rinse	11	2 Min.	130	--	11	2 Min.	60	--
Pre-Extract	--	10 Sec.	--	--	--	10 Sec	--	--
Extract	--	5 Min.	--	--	--	5 Min.	--	--

[1] Detergent, laundry, powdered, type I, MIL-D-12182 (14).

[2] Commercial cold water detergent.

TABLE III

Physical Tests Performed Prior to Long-Term Evaluations

Fabric No.	Fabric Description	Breaking Strength [1]	% Loss [2]	Tearing Strength [3]	% Loss [2]
1	Untreated "blue-line" duck	102	–	2400	–
2	"Arigal" treated "blue-line" duck	109	0	1536	36.0
3	"Statuff" treated "blue-line" duck	101	1.0	1664	30.7
4	Untreated gabardine	154	–	4900	–
5	"Arigal" treated gabardine	159	0	2256	54.0
6	"Statuff" treated gabardine	152	1.3	3050	37.8
7	Untreated "double-warp" duck	106	–	1399	–
8	"dry-cure" treated "double-warp" duck	70	34.0	732	47.7
9	"Arigal" treated "double-warp" duck	103	2.8	1305	6.7
12	Untreated OG Poplin	134	–	1606	–
13	"Arigal" treated OG Poplin	129	3.7	979	39.0
14	"Arigal" treated OG Poplin with softener	127	5.2	1011	37.0

[1] Represents mean of 10 warp specimens expressed in pounds.

[2] Based on untreated fabric.

[3] Represents mean of 5 warp specimens expressed in grams.

approximated 40%. Although the tear strength loss of fabric 9, the "Arigal" -treated "double-warp" fabric, was only 6.7%, this fabric was more soluble in cuprammonium at 82.5% than any of the other treated materials, and the excellent tear strength retention probably reflects the lack of intimate interrelationship between resin and cellulose. Fabric 13, the "Arigal"-treated OG poplin, was the only other fabric found to be substantially soluble in cuprammonium. Although the latter fabric was 29.9% soluble, it exhibited a 39.0% tear strength loss which was typical of the methylolmelamine-treated fabrics. As evidenced in fabric 14, this could not be overcome by the simple addition of a polyethylene softener to the afterwash. (Ciba, Ltd. reported a 40% tear strength loss for fabric 13 and confirmed that the loss was not reduced by the polyethylene softener.)

Ruperti (12) has suggested that the "effective" amount of resin be determined after extraction with boiling water for 1/2 hour. This treatment is reported to remove that portion of the resin which is not "fixed". Yarns teased from the "dry-cured" and "wet-cured" fabrics were refluxed with water and withdrawn at intervals up to 8 hours. These data are presented in Table IV. The nitrogen retention of fabric 6, the "Statuff"-treated gabardine fabric, after 1/2 hour of refluxing was 90.5%, but this value is not consistent with the value of 80.9% retention after 1/4 hour of leaching. The other "Statuff"-treated fabric (fabric 3 -- "Statuff"-treated "blue-line" duck) retained only 73.5% of its nitrogen following 1/2 hour of refluxing. The poorest nitrogen retention for a fabric treated by either of the other two processes was demonstrated by fabric 9, the "Arigal"-treated "double-warp" duck, which retained 80.4% of its nitrogen under the same conditions; however, since this fabric was 82.5% soluble in cuprammonium, it was not typical of the other "Arigal"-treated fabrics evaluated. Although fabric 13, the "Arigal"-treated OG poplin was 29.9% soluble, the nitrogen retention after 1 hour exposure to boiling water was still 96.8% (5). Therefore, the "Statuff" process was regarded less effective than the other two processes insofar as adequate resin fixation is concerned. After 8 hours of exposure to boiling water, all the treated fabrics lost significant amounts of resin.

Since the methylolmelamine resin treatments might be applicable to uniforms and other washables, it was important to evaluate methylolmelamine-treated fabrics for potential losses in rot-resistance due to resin losses during Army laundering. Methylolmelamine-treated fabrics were included in tests previously scheduled for other items about to be laundered. The nitrogen data are presented in Table V.

The data indicate that the methylolmelamine-treated fabrics will lose resin during laundering, but these losses can apparently be minimized by good resin fixation. Since the "Arigal"-treated fabrics were superior in nitrogen retention to the "Statuff"-treated gabardine, the results corroborate the previous data obtained from boiling the fabrics in water. Since none of the treated fabrics were subjected to cold water laundering first, it is not known whether cold water laundering at 60°F would minimize the resin losses occuring at the standard cotton wash temperatures of 100° and 130°F.

TABLE IV

Effect of Boiling Water on Nitrogen Content and % Nitrogen Retention of Yarns Teased from Resin-Treated Fabrics

Hours in Boiling Water	FABRIC DESIGNATION											
	Fabric 2 "Arigal" treated "blue line" duck		Fabric 3 "Statuff" treated "blue line" duck		Fabric 5 "Arigal" treated gabardine		Fabric 6 "Statuff" treated gabardine		Fabric 8 "dry-cure" treated "double-warp" duck		Fabric 9 "Arigal" treated "double-warp" duck	
	%N	%Ret	%N	%Ret	%N	%Ret	%N	%Ret	%N	%Ret	%N	%Ret
0	3.85	100	4.45	100	4.31	100	3.77	100	5.57	100	4.33	100
1/4	3.76	97.7	3.33	74.8	4.49	104.2	3.05	80.9	5.48	98.4	3.74	86.4
1/2	3.72	96.6	3.27	73.5	4.23	98.5	3.41	90.5	5.16	92.6	3.48	80.4
1	3.59	93.2	2.91	65.4	4.30	99.8	2.71	71.9	4.72	84.7	3.21	74.1
2	3.34	86.8	2.27	51.0	3.91	90.7	2.54	67.4	4.07	73.1	2.83	65.4
4	2.72	70.6	1.84	41.3	3.66	84.9	2.24	59.4	3.30	59.2	2.41	55.7
8	2.09	54.3	1.15	25.8	3.00	69.6	1.97	52.3	2.04	35.6	1.87	43.2

TABLE V

Effect of Laundering Tests on Nitrogen Content and % Nitrogen Retention of Resin-Treated Fabrics

	FABRIC DESIGNATION					
Laundering Tests Performed	Fabric 5 "Arigal" Treated Gabardine		Fabric 6 "Statuff" Treated Gabardine		Fabric 13 "Arigal" Treated OG Poplin	
	%N	%Ret	%N	%Ret	%N	%Ret
None	4.32	100	3.78	100	3.26	100
I std. Cotton Wash	4.25	98.4	3.32	87.8	3.27	100.3
5 std. Cotton Washes	4.13	95.6	2.57	68.0	3.19	97.9
5 std. Cotton Washes + 5 cold water Cotton Washes	4.08	94.4	2.44	64.6	3.24	99.4

Fabrics laundered 10 times are compared with the unlaundered controls in Table VI for soil burial performance. These tests were performed by U. S. Testing Company under contract. The cotton gabardine treated for maximum protection by the "Arigal" process suffered some breaking strength losses during 4 months of soil burial. Under similar circumstances, the "Statuff" control dropped precipitously despite a sufficient level of add-on.

After 10 launderings the resin-treated fabrics lost 5 to 10% breaking strength. This accounts for the subsequent lower mean breaking strength of the laundered "Arigal"-treated gabardine relative to the non-laundered control at each of the soil burial intervals. The % retention data indicate that the 10 launderings did not weaken the soil burial performance of either of the fabrics tested. The removal of approximately 1/3 of the "Statuff" resin by 10 launderings without significant loss in rot-resistance suggests that resin not covalently bonded to the cellulose plays no role in the rot-resistance of the fabric. If so, laundering should not impair the rot-resistance of the finish. Definite conclusions as to the role of chemically unbound resin in the protective mechanism cannot be formulated until a more exhaustive series of laundering tests is performed.

b. Soil Burial

The extended soil burial and outdoor weathering data were obtained largely from white fabrics exposed in two separate studies. In the earlier study, "blue-line" duck and gabardine fabrics prepared by the two "wet-cure" processes were compared side by-side for differences in long-term weather- and rot resistance between treatments. As a result of this work, a later study was initiated to compare the "Arigal" process, the better of the two "wet-cure" processes, with the "dry-cure" process of Berard under identical test conditions on the same "double-warp" duck fabric. For comparative purposes the data from the two studies will be considered together. Fabric 13, the "Arigal"-treated OG poplin, which contained 8.0% resin, is also included because it was the only other fabric subjected to extended soil burial and outdoor weathering.

Table VII demonstrates the effect of soil burial on the breaking strength of methylolmelamine-treated fabrics prepared by the three processes. The data for "blue-line" fabrics 2 and 3 can be compared directly with soil burial data previously reported for the "dry-cure" fabric discussed by Kempton *et al* (6) since the base fabric and soil burial conditions were identical. "Double-warp" fabrics 8 and 9 were removed from soil burial at bi-monthly rather than monthly intervals because of durability of the treatments involved. Fabric 3, the "Statuff"-treated "blue-line" duck, performed somewhat better than the "Statuff"-treated gabardine previously discussed but no better than the "add-on" fungicides already in military use. The "Arigal"-treated "blue-line" fabric performed approximately as well as the "Arigal"-treated gabardine during 3 months soil burial; its performance during the fourth month, however, was somewhat inferior – 65.1% retention relative to 88.8% for the "Arigal"-treated gabardine. It is evident that both of these

TABLE VI

Effect of Laundering on the Soil Burial Performance of Fabrics Laundered 10 Times[1]

Days Soil Burial	FABRIC DESIGNATION: Fabric 5 "Arigal" Treated Gabardine — No Laundering: B.S. in Lbs.	% Ret.	Laundered 10X: B.S. in Lbs.	% Ret.	Fabric 6 "Statuff" Treated Gabardine — No Laundering: B.S. in Lbs.	% Ret.	Laundered 10X: B.S. in Lbs.	% Ret.
0	183.5	100	164.8	100	169.5	100	160.5	100
30	178.4	97.2	162.4	98.5	100.3	59.2	133.8	83.4
60	176.0	95.9	157.0	95.3	25.8	15.2	36.3	22.6
90	170.0	92.6	157.0	95.3	19.3	11.4	...[2]	...[2]
120	163.0	88.8	146.0	88.6	...[2]	...[2]	...[2]	...[2]

[1] Five standard cotton washes and five cold water launderings.

[2] Samples destroyed during soil burial.

TABLE VII

Effect of NLABS Soil Burial on the Breaking Strength of Methylolmelamine Treated Fabrics Prepared by Three Different Processes

	FABRIC DESIGNATION									
Months Soil Burial	Fabric 2 "Arigal" Treated "blue-line" Duck		Fabric 3 "Statuff" Treated "blue-line" Duck		Fabric 8 "Dry-cure" Treated "double-warp" Duck		Fabric 9 "Arigal" Treated "double-warp" Duck		Fabric 13 "Arigal" Treated OG Poplin	
	B.S. in lbs.	%Ret.	B.S. in lbs.	%Ret.	B.S. in lbs.	%Ret.	B.S. in lbs.	%Ret.	B.S. in lbs.	%Ret.
0	109	100	101	100	69.6	100	103.3	100	129	100
1	107	98.2	105	104.0					129	100
2	110	100.9	87	86.1	85.6	123.0	101.8	98.5	130	100.8
3	106	97.2	38	37.6					130	100.8
4	71	65.1	13	12.9	83.3	119.7	97.7	94.6	130	100.8
5	60	55.0							124	96.1
6	42	38.5			83.4	119.8	98.2	95.1	118	91.5
7	41	37.6							107	82.9
8	47	43.1			82.2	118.1	87.9	85.1	92	71.3
9	40	36.7							59	45.7
10	38	34.9			89.0	127.9	86.7	83.9	40	31.0
11	38	34.9							27	20.9
12	27	24.8			86.0	123.6	90.4	87.5	32	24.8
13	24	22.0							20	15.5
14	18	16.5			83.3	119.7	68.6	66.4	19	14.7
16					58.3	83.8	51.5	49.9		

"Arigal"-treated fabrics significantly outperformed either of the "Statuff"-treated fabrics in soil burial. The weaker performance of the "Statuff"-treated "blue-line" fabric relative to the "Arigal"-treated "blue-line" fabric cannot be attributed to resin content alone since their relative nitrogen contents were 4.37% and 3.94%, respectively. The inferior soil burial performance of fabrics prepared by the "Statuff" process is evidence that rot-resistance cannot be gauged by resin content alone, that the protective mechanism must be related to the nature of the "resin-cellulose complex" which results from the treatment process.

Fabric 9, the "Arigal"-treated "double-warp" duck, was superior in rot-resistance to previous "Arigal"-treated fabrics evaluated in in-house tests and almost the equal of fabric 8, the "dry-cure"-treated "double-warp" fabric, which showed slightly greater longevity in soil burial. Although the breaking strength data for the soil buried samples indicate a significant difference when based as a percentage on the after-treatment value, the raw breaking strength data do not indicate the difference in performance between the two treated fabrics to be quite as large. For example, after 16 months of soil burial the mean breaking strengths of the "dry cure" and "Arigal"-treated "double-warp" specimens were 58.3 and 51.5 pounds, respectively, but these values correspond to percentages of 83.8 and 49.9 – figures that are more disparate than the raw breaking strength data. Since the loss of breaking strength following "dry-cure" treatment was partially recovered during subsequent soil burial, the "dry-cure" treated fabric benefits unfairly when soil burial data are compared as percentages based on the after-treatment, no soil burial value, because the lower basis value minimizes the breaking strength changes occurring during soil burial.

The loss of breaking strength following "dry-cure" treatment and subsequent "regain" during soil burial have been discussed by Berard (1) and Kempton (6). Presumably the breaking strength "regain" represents the breaking of cross-links which are formed during the fixation of resin by the "dry-cure" process. Since the "dry-cured" fabric discussed by Kempton regained its original breaking strength, there was no microbiological deterioration until the cross-links, which had cause the initial breaking strength loss, were broken after 64 weeks of burial. In the current study the "dry-cure" treated "double-warp" fabric did not regain its original breaking strength of 106 pounds. This could indicate either that the base fabric had been permanently damaged during the treatment process or that there was microbiological degradation occurring simultaneously with the breaking strength regain. Visual observation, however, indicated that microbiological degradation had occurred prior to 6 months of soil burial. Therefore, the rot-resistance of the "dry-cure" treated "double-warp" duck did not equal the rot-resistance of the fabric described by Kempton (6).

Table VIII contains cuprammonium solubility data derived from fabrics 2, 3, 8, 9 and 13 prior to and after soil burial. Cuprammonium insolubility of resin-treated cellulose has been regarded by some workers as indicative of cross-linking between the cellulose and the resin. Mehta and Mehta, however, have discussed evidence that cross-linking is

TABLE VIII

Effect of NLABS Soil Burial on the Solubility in Cuprammonium of Methylolmelamine Treated Fabrics Prepared by Three Processes

	FABRIC DESIGNATION									
Months Soil Burial	Fabric 2 "Arigal" Treated "blue-line" Duck		Fabric 3 "Statuff" Treated "blue-line" Duck		Fabric 8 "dry-cure" Treated "double-warp" Duck		Fabric 9 "Arigal" Treated "double-warp" Duck		Fabric 13 "Arigal" treated OG Poplin	
	%Sol.	Δ %Sol.[1]	%Sol.	Δ %Sol.[1]	%Sol.	Δ %Sol.[1]	%Sol.	Δ %Sol.[1]	%Sol.	Δ %Sol.[1]
0	3.4	0	3.0	0	2.9	0	82.5	0	29.9	0
1	6.5	3.1	0.9	2.1					33.3	3.4
2	5.1	1.7	6.1	3.1	13.1	10.2	89.6	7.1	36.1	6.2
3	11.0	7.6	6.8	3.8					42.6	12.7
4	9.4	6.0	10.9	7.9	21.2	18.3	96.3	13.8	46.0	16.1
5	5.8	2.4							45.7	15.8
6	14.8	11.4			21.1	18.2	96.3	13.8	62.0	32.1
7	19.9	16.5							62.2	32.3
8	21.8	18.4			29.3	26.4	96.6	14.1		
9	37.5	34.1							69.7	39.8
10	22.5	19.1			30.9	28.0	97.5	15.0	70.6	40.7
11	22.0	18.6							68.7	38.8
12	23.9	20.4			35.3	32.4	98.7	16.2	71.6	41.7
13									70.2	40.3
14					37.0	34.1	100.0	17.5	71.3	41.4
16					42.0	39.1				

[1] Change in % solubility with increasing intervals of soil burial.

not essential for insolubility and have concluded that steric hindrance or strong hydrogen bond formation are sufficient to prevent coordination between $Cu(NH_3)_4$ and four hydroxyls in the 2 and 3 positions of adjacent cellulose chains (9). Thus solubility data should be supported by other independent data when making judgments regarding cross-linking. It would be questionable to assume that fabrics 2, 3 and 8 treated by three different processes were all similar highly cross-linked systems because of their comparable low solubility in cuprammonium.

Following methylolmelamine resin treatment cotton fabrics were generally found to be cuprammonium-insoluble regardless of the process involved. Fabric 9, the "Arigal"-treated "double-warp" duck, and fabric 13, the "Arigal"-treated OG poplin, were exceptions, however. Since fabric 9 contained as much or more resin than other fabrics which were essentially insoluble, there was enough resin in the fabric to have produced an insoluble product. The 82.5% solubility of fabric 9 in cuprammonium may be related to the relatively poor resin retention of this fabric following extraction with boiling water, which has been previously discussed. Although these data indicated that this fabric was not typical of the other fabrics prepared by the "Arigal" process, this fabric retained 94.6% of its breaking strength after 4 months of exposure to soil. Therefore, a high degree of insolubility in cuprammonium or boiling water cannot be regarded strictly essential for good rot-resistance. Furthermore, since both "Statuff"-treated fabrics were highly cuprammonium-insoluble but failed after 1-2 months of soil burial, cuprammonium insolubility cannot even be regarded as assurance of soil burial protection.

The changes occurring in the cuprammonium solubility of resin-treated cellulose during prolonged soil burial offer further evidence that this measurement is not directly related to rot-resistance. Changes in the cuprammonium solubility of fabrics prepared by the three different processes were fairly similar in relation to the wide dissimilarities in the microbiological resistance of the fabrics as evidenced by their breaking strength retentions. For example, after 4 months of soil burial, the breaking strengths of the five fabrics ranged from a low of 12.9% to a high of 119.7% retention, but the changes in % solubility only ranged from 6.0% to 18.3% for the five fabrics. Furthermore, the cuprammonium solubilities of fabrics which had failed in soil burial were markedly different – 10.9% for the "Statuff"-treated "blue-line" duck, 23.8% for the "Arigal"-treated "blue-line" duck and 71.3% for the "Arigal"-treated OG poplin. But the "Arigal"-treated "double-warp" duck was not markedly susceptible to microorganisms even when 100% soluble in cuprammonium since the breaking strength retention was only reduced from 66.4 to 49.9% between 14 and 16 months of soil burial. Cuprammonium solubility as a measure of change in the "resin-cellulose complex" during prolonged soil burial is indicating an overall change in the complex which is not directly related to the protective mechanism.

Table IX contains nitrogen content data derived from fabrics 2, 3, 8, and 9 prior to and after soil burial. As previously discussed, the soil burial performance of these fabrics was dependent on the physico-chemical relationship of the resin to the cellulose rather than on the actual differences in resin content resulting from treatment. Likewise, the changes in nitrogen content which occurred during soil burial bore little relationship

TABLE IX

Effect of NLABS Soil Burial on the Nitrogen Content and % Nitrogen Retention of Methylolmelamine Treated Fabrics Prepared by Three Different Processes

	FABRIC DESIGNATION									
Months Soil Burial	Fabric 2 "Arigal" Treated "blue-line" Duck		Fabric 3 "Statuff" Treated "blue-line" Duck		Fabric 8 "dry-cure" Treated "double-warp" Duck		Fabric 9 "Arigal" Treated "double-warp" Duck		Fabric 13 "Arigal" treated OG Poplin	
	%N	%Ret.	%N	%Ret.	%N	%Ret.	%N	%Ret.	%N	%Ret.
0	3.94	100	4.37	100	5.32	100	4.32	100	3.27	100
1	4.11	104.3	4.07	93.1					3.26	99.7
2	4.24	107.6	4.02	92.0	5.03	94.5	4.42	102.3	3.30	100.9
3	3.98	101.0	4.66	106.6					3.22	98.5
4	3.82	97.0	5.19	118.8	4.65	87.4	3.72	86.1	3.20	97.9
5	4.16	105.6							3.18	97.2
6	4.38	111.2			4.38	82.3	3.25	75.2	3.17	96.9
7	4.37	110.9							3.18	97.2
8	4.10	104.1			4.30	80.8	3.17	73.4		
9	4.17	105.8							3.30	100.9
10	4.15	105.3			4.05	76.1	3.10	71.8	3.34	102.1
11	4.29	108.9							3.58	109.5
12	4.41	111.9			3.79	71.2	2.85	66.0	3.40	104.0
13									3.84	117.4
14					3.48	65.4	2.81	65.0	3.75	114.7
16					3.45	64.8	2.75	63.7		

to the relative fungal susceptibilities of the fabrics. After 4 months of soil burial performed in-house, the breaking strengths of the four fabrics ranged from a low of 12.9% to a high of 119.7% retention, but the nitrogen retentions only ranged from 86.1% to 118.8%. When soil burial was terminated, the fabrics retained 63.7% to 118.8% of their original nitrogen content. This is further evidence that emphasis sould be placed on the qualitative relationship of the resin to the cellulose rather than on the quantitive one. At present, there are no chemical techniques that adequately define the deterioration of resin-treated fabrics in soil burial.

c. Outdoor Weathering

The breaking strength, cuprammonium solubility and nitrogen content data for the wet- and dry-cured fabrics exposed outdoors 2 years were obtained from three separate exposures. Fabrics 4-6 (7.2 oz. bleached white gabardine) were exposed from September 1962 to September 1964, fabrics 12 and 13 (6 oz. OG poplin) from May 1963 to May 1965 and fabrics 7-9 (9.85 oz. bleached white "double-warp" duck) from June 1965 to June 1967. Comparisons between the three exposures were based on 12 and 24-month intervals, whenever possible, to eliminate seasonal variations.

Table X demonstrates the effect of outdoor exposure on the breaking strength of untreated and methylolmelamine-treated fabrics prepared by the three processes. All three processes protected the cotton base fabric from actinic degratation. After a year of outdoor weathering, the % breaking strength retentions of the untreated fabrics ranged from a low of 21.6% for the OG poplin to a high of 39.6% for the white gabardine material. The % retentions for the treated fabrics during the same interval ranged from a low of 59.7% for the "Arigal"-treated OG poplin to a high of 96.6% for the "dry-cure"-treated "double-warp" duck. The next highest % retention level at the 12-month interval was demonstrated by the "Statuff"-treated gabardine which retained 90.1% of its strength. Although outdoor exposure of the untreated fabric had to be terminated after 12 to 14 months, it was possible to weather all the methylolmelamine-treated fabrics for a full 2 years. In contrast, fabrics treated with the conventional "add-on" fungicides in military use will not weather longer than a year in this temperate climate. After 24 months outdoors the methylolmelamine-treated fabrics ranged from a low of 29.5% retention for the "Arigal"-treated OG poplin to a high of 71.0% for the "dry-cure"-treated "double-warp" duck. Since the "Statuff"-treated gabardine fabric retained 67.8% of its strength following the full 2 years exposure, it was apparent that the "Statuff" process could not be faulted for the degree of weather-resistance it provided. All three processes, in effect, provided excellent protection. The data from the "Arigal"-treated "double-warp" duck, which was 82.5% soluble in cuprammonium following treatment, was evidence that a high degree of cuprammonium insolubility was not essential for good weather-resistance.

Table XI demonstrates the effect of outdoor exposure on the cuprammonium fluidity of control fabrics and cuprammonium solubility of methylolmelamine-treated fabrics prepared by the three processes. All the treated fabrics were essentially cuprammonium-insoluble following methylolmelamine treatment with the exception of the

TABLE X

Effect of Outdoor Exposure at the NLABS Sudbury Annex on the Breaking Strength of Untreated and Methylolmelamine Treated Fabrics Prepared by Three Different Processes

Months Outdoor Exposure	Fabric 4 Untreated Gabardine B.S. in lbs	% Ret	Fabric 5 "Arigal" Treated Gabardine B.S. in lbs.	% Ret	Fabric 6 "Statuff" Treated Gabardine B.S. in lbs.	% Ret	Fabric 7 Untreated "double-warp" Duck B.S. in lbs.	% Ret	Fabric 8 "Dry-cure" Treated "double-warp" Duck B.S. in lbs.	% Ret	Fabric 9 "Arigal" Treated "double-warp" Duck B.S. in lbs.	% Ret	Fabric 12 Untreated OG Poplin B.S. in lbs.	% Ret	Fabric 13 "Arigal" Treated OG Poplin B.S. in lbs.	% Ret
	Initiated September 1962								Initiated June 1965				Initiated May 1963			
0	154	100	159	100	152	100	106.5	100	69.6	100	103.3	100	134	100	129	100
2	144	93.5	165	103.8	163	107.2	60.8	57.1	74.7	107.3	91.7	88.8	97	72.4	119	92.2
4	147	95.5	171	107.5	171	112.5	49.3	46.3	70.6	101.4	88.8	86.0	72	53.7	107	82.9
6	132	85.7	167	105.0	163	107.2	48.7	45.7	69.8	100.3	83.7	81.0	61	45.5	95	73.6
8	127	82.5	156	98.1	168	110.5	45.1	42.3	71.3	102.4	82.2	79.6	51	38.1	92	71.3
10	87	56.5	149	93.7	154	101.3	34.9	32.8	69.4	99.7	75.6	73.2	39	29.1	83	64.3
12	61	39.6	134	84.3	137	90.1	32.3	30.3	67.2	96.6	72.2	69.9	29	21.6	77	59.7
14	33	21.4	135	84.9	135	88.8	17.6	16.5	61.0	87.6	66.1	64.0	Terminated 5/1964		68	52.7
16	Terminated 11/1963		136	85.5	126	82.9	Terminated 8/1966		55.8	80.2	59.0	57.1			61	47.3
18			135	84.9	129	84.9			51.9	74.6	61.4	59.4			49	38.0
20			114	71.7	118	77.6			54.6	78.4	59.5	57.6			48	37.2
22			115	72.3	89	58.6			51.5	74.0	59.3	57.4			48	37.2
24			94	59.1	103	67.8			49.4	71.0	58.1	56.2			38	29.5
			Terminated 9/1964		Terminated 9/1964				Terminated 6/1967		Terminated 6/1967				Terminated 5/1965	

TABLE XI

Effect of Outdoor Exposure at the NLABS Sudbury Annex on the Fluidity and Solubility in Cuprammonium of Untreated and Methylolmelamine Treated Fabrics Prepared by Three Processes

	FABRIC DESIGNATION															
Months	Fabric 4 Untreated Gabardine		Fabric 5 "Arigal" Treated Gabardine		Fabric 6 "Statuff" Treated Gabardine		Fabric 7 Untreated "double-warp" Duck		Fabric 8 "Dry-cure" Treated "double-warp" Duck		Fabric 9 "Arigal" Treated "double-warp" Duck		Fabric 12 Untreated OG Poplin		Fabric 13 "Arigal" Treated OG Poplin	
Outdoor Exposure	Fluidity in Rhes	Δ Rhes[1]	% Sol	Δ% Sol[1]	% Sol	Δ% Sol[1]	Fluidity in Rhes	Δ Rhes[1]	% Sol	Δ% Sol[1]	% Sol	Δ% Sol[1]	Fluidity in Rhes	Δ Rhes[1]	% Sol	Δ% Sol[1]
	Initiated 9/1962								Initiated 6/1965				Initiated 5/1963			
0	2.4	0	2.0	0	1.3	0	23.5	0	2.9	0	82.5	0	5.0	0	29.9	0
2	13.7	11.3	20.6	18.6	17.1	15.8	38.8	15.3	28.7	25.8	92.2	9.7	25.9	20.9	91.9	62.0
4	16.7	14.3	27.2	25.2	23.2	21.9	41.6	18.1	40.5	37.6	96.2	13.7	34.0	29.0	97.6	67.7
6	19.2	16.8	44.4	42.4	43.0	41.7	42.1	18.6	49.6	46.7	97.7	15.2	36.9	31.9	98.3	68.4
8	27.6	25.2	69.1	67.1	81.2	79.9	40.8	17.3	52.5	49.6	99.3	16.8	37.1	32.1	98.2	68.3
10	34.8	32.4	91.6	89.6	97.2	95.9	45.5	22.0	57.4	54.5	97.7	15.2	40.1	35.1	98.2	68.3
12	41.9	39.5	97.3	95.3	98.4	97.1	Terminated 8/1966		78.9	76.0	99.0	16.5	42.5	37.5	98.5	68.6
14	43.7	41.3	98.7	96.7	98.2	96.9			95.8	92.9	99.3	16.8	Terminated 5/1964		98.6	68.7
16	Terminated 11/1963		99.4	97.4	99.3	98.0			97.5	94.6	99.0	16.5			98.9	68.9
18			98.9	96.9	99.6	98.3			96.5	93.6	99.0	16.5			Terminated 5/1965	
20			99.5	97.5	99.8	98.5			98.0	95.1	99.5	17.0				
22			99.8	97.8					99.3	96.4	99.5	17.0				
24			Terminated 9/1964						99.6	96.7	99.5	17.0				
									Terminated 6/1967							

[1] Change in rhes or % solubility with increasing intervals of outdoor exposure.

two "Arigal"-treated fabrics already noted – fabric 9, the "double-warp" duck, and fabric 13, the OG poplin. Prior to any exposure, the cuprammonium fluidities of the untreated gabardine, untreated OG poplin and untreated "double-warp" duck were 2.4, 5.0 and 23.5 rhes, respectively. Faulty preparation of the "double-warp" duck, which resulted in damage to the fabric, manifested itself as a high fluidity of 23.5 rhes. However, like the other control fabrics, the "double-warp" duck retained sufficient strength to last 12-14 months outdoors, and following either "Arigal" or "dry-cure" treatment the "double-warp" duck weathered as well as any of the other treated fabrics.

The soil burial of all the treated fabrics was terminated prior to their conversion to a product completely soluble in cuprammonium. However, during weathering, all the treated fabrics became essentially 100% soluble in cupramonnium prior to termination of the test. Typically the fabrics were readily converted by weathering to a product that was 95 to 97% soluble in cuprammonium prior to the loss of much breaking strength. For example, when 95-97% soluble in cuprammonium, the "Arigal"-treated gabardine retained 84% of its strength, the "Statuff"-treated gabardine 101%, the "dry-cure"-treated "double-warp" duck 88%, the "Arigal"-treated "double-warp" duck 86% and the "Arigal"-treated OG poplin 83%. Therefore, cuprammonium solubility measurements were regarded as a highly sensitive index of weathering effects. Conversion of the last 5% of the insoluble resin-treated cellulose to a cuprammonium-soluble product proceeded with difficulty and required at least an additional 6 months or more of weathering. The "Arigal"-treated OG poplin demonstrated the greatest 2-month increase in cuprammonium solubility for all fabrics – a 62.0% change between 0 and 2 months of outdoor exposure.

It was postulated by Kempton *et al* (6) that crosslinks were being broken during the outdoor exposure of "dry-cured" duck with the resultant regain of the breaking strength loss incurred on treatment. His view was supported by measurement of the cuprammonium fluidity after the fabric had become 100% soluble in cuprammonium. This indicated 50% degradation rather than the net 30% breaking strength loss observed on termination of the exposure after 22 months. Cuprammonium fluidity measurements of both "wet-cured" gabardine fabrics after 24 months exposure indicated approximately 40% deterioration, and the observed breaking strength loss for both fabrics averaged 37% after 24 months exposure. The agreement between breaking strength and cuprammonium fluidity data from the gabardine fabrics supports the hypothesis that the observed breaking strength loss of only 30% in the "dry-cured" duck was due to release of crosslinks during outdoor exposure. Therefore, cuprammonium fluidity measurements are more reliable than breaking strength losses as an index of the degradation resulting from weathering, particularly, in the case of crosslinked systems where actinic degradation can be masked by regain of the breaking strength loss incurred on treatment.

Table XII demonstrates the effect of outdoor exposure on the nitrogen content and % nitrogen retention of methylolmelamine-treated fabrics prepared by the three processes. Following methylolmelamine treatment the fabrics ranged from 3.27 to 5.32% in nitrogen content. Changes in nitrogen content, unlike cuprammonium solubility changes, occurred throughout the entire 2 years of outdoor exposure. Since the 62% increase in the

TABLE XII

Effect of Outdoor Exposure at the NLABS Sudbury Annex on the Nitrogen Content and % Nitrogen Retention of Methylolmelamine Treated Fabrics Prepared by Three Processes

Months Outdoor Exposure	Fabric 5 "Arigal" Treated Gabardine		Fabric 6 "Statuff" Treated Gabardine		Fabric 8 "Dry-cure" Treated "double-warp" duck		Fabric 9 "Arigal" Treated "double-warp" duck		Fabric 13 "Arigal" Treated OG Poplin	
	% N	% Ret	% N	% Ret	% N	% Ret	% N	% Ret	% N	% Ret
	Initiated 9/1962				Initiated 6/1965				Initiated 5/1963	
0	4.31	100	3.77	100	5.32	100	4.32	100	3.27	100
2	4.73	109.7	3.38	89.7	5.03	94.5	3.48	80.6	2.76	84.4
4	4.46	103.5	3.34	88.6	4.34	81.6	3.30	76.4	2.22	67.9
6	4.49	104.2	3.24	85.9	4.55	85.5	3.15	72.9	1.82	55.7
8	4.34	100.7	2.91	77.2	4.19	78.8	2.53	58.6	1.67	51.1
10	3.76	87.2	2.40	63.7	4.48	84.2	2.69	62.3	1.55	47.4
12	3.04	70.5	2.01	53.3	3.63	68.2	2.18	50.5	1.40	42.8
14	2.78	64.5	2.05	54.4	3.08	57.9	1.65	38.2	1.08	33.0
16	2.58	59.9	1.81	48.0	2.53	47.6	1.42	32.9	0.91	27.8
18	2.54	58.9	1.64	43.5	2.42	45.5	1.38	31.9	0.74	22.6
20	2.29	53.1	1.77	46.9	2.38	44.7	1.33	30.8	0.72	22.0
22	1.94	45.0	1.44	38.2	2.23	41.9	1.30	30.1	0.69	21.1
24	1.52	35.3	1.37	36.3	1.87	35.2	1.30	30.1	0.65	19.9
	Terminated 9/1964				Terminated 6/1967				Terminated 5/1965	

cuprammonium solubility of the "Arigal"-treated OG poplin following 2 months of outdoor exposure was not associated with a large loss of nitrogen content, changes in the resin-cellulose relationship detectable by solubility measurements can remain undetected by nitrogen measurements. The relatively weak breaking strength retention of this fabric during outdoor exposure could be related to its 3.27% nitrogen content (8.0% resin) following treatment since all the other fabrics contained 1% or more resin than it and weathered better. Although the "Arigal"-treated OG poplin was 29.9% soluble in cuprammonium prior to weathering, the "Arigal"-treated "double-warp" duck at 82.5% solubility in cuprammonium weathered well. Therefore, the lack of cuprammonium insolubility *per se* could not be faulted for the weaker weather-resistance of the "Arigal"-treated OG poplin relative to the other fabrics. The weak resistance of the "Statuff" finish to either boiling water or laundering manifested itself as a rapid fall-off in the nitrogen content of the "Statuff"-treated gabardine relative to the "Arigal"-treated gabardine, which was exposed in parallel with it. This decline in nitrogen content corresponded to the fall-winter season during which the "Arigal"-treated gabardine lost none of its nitrogen content. However, it should be noted that the loss in nitrogen did not have an adverse effect on the good weather-resistance of the "Statuff"-treated fabric as determined by breaking strength measurements.

In our experience, cuprammonium fluidity measurements performed on control fabrics exposed outdoors at the NLABS Sudbury Annex have always indicated the degradation to be primarily actinic. Nevertheless, all resin-treated materials have shown evidence of the non-destructive growth of both algae and fungi during outdoor weathering. In the soil burial studies, it was noted that surface growth could not be washed off only after structural damage had occurred. Therefore, such growth should be no problem in launderable items. Other material may require the use of a biocide.

The weathered fabrics are compared in Table XIII for their relative abilities to withstand microbiological attack after increasing intervals of actinic exposure. Soil burial data for the non-weathered fabrics where available were included as a basis for comparing the loss of rot-resistance due to weathering. Comparisons between the fabrics should be based on 12 and 24-month intervals of outdoor exposure to eliminate seasonal differences between the three different exposures. However, direct comparison at any given interval of exposure can be made between treatments on the same base fabric since a different base fabric was employed for each of the exposures. The performance of the "Statuff"-treated gabardine during soil burial following outdoor exposure was inferior relative to the good performance demonstrated by the gabardine and "double-warp" duck fabrics treated by either the "Arigal" or "dry-cure" process. The poor performance of the "Arigal"-treated OG poplin is further evidence that this fabric was not representative of the other "Arigal"-treated fabrics. Two months of outdoor exposure resulted in a 62% increase in cuprammonium solubility and a marked loss in rot resistance. The "Arigal" and "dry-cure" treatments both performed well on the "double-warp" duck during identical exposure conditions as did the "Arigal"-treated gabardine fabric during a previous exposure. Therefore, both treatments should be equally capable of protecting the base fabric from microbiological damage during actinic exposure.

TABLE XIII

% Retention of Breaking Strength Following NLABS Soil Burial for 1-3 Months of Weathered Methylolmelamine Treated Fabrics Prepared by Three Different Processes [1]

Preliminary Outdoor Exposure in Months	FABRIC DESIGNATION: Fabric 5 "Arigal" Treated Gabardine			Fabric 6 "Statuff" Treated Gabardine			Fabric 8 "Dry-cure" Treated "double-warp" duck			Fabric 9 "Arigal" Treated "double-warp" duck			Fabric 13 "Arigal" Treated OG Poplin		
	1 mo S.B.	2 mo S.B.	3 mo S.B.	1 mo S.B.	2 mo S.B.	3 mo S.B.	1 mo S.B.	2 mo S.B.	3 mo S.B.	1 mo S.B.	2 mo S.B.	3 mo S.B.	1 mo S.B.	2 mo S.B.	3 mo S.B.
0								123			99		100	100	101
2			102			85			101			93	63		2
4			97			49			98			81	48	7	0
6			96		45				91			83	18		
8			96	73					76			79	17		
10			72	34					84			67			
12		46		0				95			96				
14	76							93			64				
16	88							37			46				
18	96							46			10				
20	98						94			10[illegible]					
22	89						101			91					
24	86						70			33					

[1] Based on unburied, weathered, treated fabrics.

d. Performance at Lower Add-On Levels

Several typical Army fabrics were treated by the "Arigal" process to contain 7% total add-on to determine if that level would perform equivalent to the best add-on fungicides available. Fabrics were prepared as small lab batches and tested under our screening program. Standard soil burial was performed before and after leaching and 100 hours of weatherometer exposure. These tests were performed on a 6 oz. dyed poplin and 8.8 oz. vat-dyed sateen containing 7.74 and 7.32% resin, respectively, by nitrogen analysis.

Rot-resistance data prior to and after leaching and exposing fabrics 10 and 11 for 100 hours in a weatherometer are contained in Table XIV. The rot-resistance of both fabrics prior to and after leaching was very good and comparable to the "Arigal"-treated fabrics containing higher resin add-ons. Following 100 hours of weatherometer exposure, the 6 oz. poplin lost 18.9% of its breaking strength but very little of its rot-resistance. The results obtained from this fabric were unlike those obtained from fabric 13, the 6 oz. poplin which, following only a 7.8% breaking strength loss after 2 months of outdoor exposure, retained only 63% of its strength after a month of subsequent soil burial. In effect, "Arigal"-treated fabrics containing either 7.74 or 7.32% resin out-performed fabric 13 which contained 8.0% resin. Therefore, the inferior performance of fabric 13 was probably due to faulty preparation of the base fabric or faulty application of the treatment rather than insufficient resin. However, since it is undoubtedly easier to maintain careful quality control in small laboratory batches than in larger plant runs, a higher level of resin add-on may be required for a margin-of-safety.

4. Conclusions

Methylolmelamine resin treatments provide several fold better weather- and rot-resistance for cotton textiles than can be achieved by conventional add-on fungicides. However, the resin-treated fabrics, as supplied, can be variable in the desired biological and physical properties despite the supervision of personnel experienced in the respective processes and application according to the best available practice.

Such variability was most keenly reflected in the rot-resistance of these fabrics. The most rot-resistant methylolmelamine-treated fabric evaluated by this laboratory has been a "dry-cured" duck which endured 64 weeks of soil burial with no breaking strength losses (6). In the subsequent side-by-side comparison between the "dry-cure" and "Arigal" processes on the same base fabric, the "dry-cure" process again demonstrated its excellence in soil burial. However, the difference in rot-resistance between the processes was not as large as anticipated - partially because the "Arigal"-treated fabric outperformed any of the others previously evaluated in-house and the "dry-cured" fabric did not equal its predecessor. The "Staturf"-treated fabrics evaluated were judged inferior rather than variable in soil burial performance on the basis of the tests performed. The weak soil burial performance of "Statuff"-treated fabrics may not be directly related to the lack

TABLE XIV

Soil Burial Data from Fabrics 10 and 11 Prior To and After Leaching and 100 Hours of Weatherometer Exposure as Performed by U. S. Testing Company Under Contract

Tests Performed	FABRIC 10 "Arigal" Treated 6 oz. Poplin Containing 7.74% Resin		FABRIC 11 "Arigal" Treated 8.8 oz. Sateen Containing 7.32% Resin	
	B.S. in lbs.	% Ret	B.S. in lbs.	% Ret
Original Fabric, as received	114.7	100	137.0	100
Original, 1 mo. soil burial	112.3	98.0	141.0	102.9
2 mo. soil burial	112.3	98.0	133.8	97.7
3 mo. soil burial	112.0	97.6	134.3	98.0
4 mo. soil burial	103.8	90.5	124.8	91.1
Leached	119.7	104.4[1]	135.3	98.8[1]
Leached, 1 mo. soil burial	115.3	96.3	141.3	104.4
2 mo. soil burial	111.0	92.7	127.5	94.2
3 mo. soil burial	111.5	93.1	133.0	98.3
4 mo. soil burial	107.0	89.4	81.5	60.2
Weatherometer	93.0	81.1[1]	126.6	92.4[1]
Weatherometer, 1 mo. soil burial	86.0	92.5	133.3	105.3
2 mo. soil burial	86.0	92.5	124.5	98.3
3 mo. soil burial	76.5	82.3	122.5	96.8
4 mo. soil burial	73.5	79.0	121.0	95.6

[1] These % retention values based on original fabric, as received.

of adequate resin fixation discerned from the laundering and boiling water experiments since the "Arigal"-treated "double-warp" duck, which also demonstrated a weakness to boiling water, performed well in the soil burial test. Therefore, the poor rot-resistance of "Statuff"-treated fabrics could be due to some inherent weakness of the process or to the use of the methylated form of the compound. The laundering tests also indicate that methylolmelamine-treated fabrics can be laundered 10 times without any loss in microbiological resistance, and items laundered regularly should not require the use of a biocide for control of surface growth because non-destructive growth should be readily washed off.

All the treated fabrics weathered excellently regardless of process or significant differences in microbiological resistance. The "Arigal"-treated OG poplin which did not weather quite as well as the other fabrics during extended outdoor weathering contained only 8.0% resin relative to other fabrics containing 9-12% resin.

Outdoor exposure followed by soil burial is the best overall basis for comparison of the finishes since this test is more typical of field conditions. In this regard, the "Arigal" finish was comparable to the "dry-cure" finish. After 12 months of weathering, "Statuff" finished cotton could not survive a month of soil burial testing. According to Boyle (2), the "Statuff" process has been reformulated, but any recommendation of the new finish must be based on the outcome of long-term environmental testing.

The selection of a particular finish must ultimately be made on the basis of the field conditions anticipated and the intended usage of the item. The "Arigal" finish should be suitable for field conditions resulting in a combination of actinic and microbiological degradation. If fiber stiffness and lowered breaking strength can be tolerated, the "dry-cure" finish as proposed by Berard (1) can also be considered. The good rot- and weather-resistance data from "Arigal"-treated "double-warp" duck was evidence that methylolmelamine resin treatments did not have to result in serious damage to the mechanical properties of the fabric and that cuprammonium insolubility was not a prerequisite for good field performance. If the small tear strength loss measured in this fabric resulted from a basic modification in the "Arigal" process rather than a stroke of luck, then the objection of textile technicians to resin treatments because of the mechanical damage caused by them would no longer be valid.

5. References

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APPENDIX

Annual Climatological Data for NLABS Sudbury Annex – 1962

Month	WIND[1] Prevailing Dir. 2mtr	Average Hourly Speed 50 cm	Average Hourly Speed 2mtr	Maximum Hourly Average Speed 50cm	Maximum Hourly Average Day	Maximum Hourly Average Dir. 2mtr	Maximum Hourly Average Speed 2mtr	Maximum Hourly Average Day	SOLAR RADIATION (Langleys/Day) All Wave Total Hem.	All Wave Net Ex.	Vertical Eppley 2mtr	Filtered 45° Eppley Clear	Yellow	Lt Red	Dk Red
JAN	SW	2	2	7	23	WNW	10	16	649	-82	––	318	––	217	191
FEB	NNE	1	2	5	6	NE	9	15	––	––	211	314	264	200	183
MAR	N	2	4	7	24	N	11	24	980	102	382	475	365	298	254
APR	NW	2	3	8	24	N	11	24	––	123	411	435	325	271	226
MAY	NE	1	2	7	10	NNE	11	9	––	207	489	396	335	––	223
JUN	NE	––	1	––	––	SSW	4	2	1306	240	––	––	355	––	245
JUL	NNW	––	2	––	––	N	8	2	1300	228	––	––	348	––	250
AUG	N	––	2	––	––	N	9	29	1186	151	402	437	345	274	247
SEP	WNW	1	2	7	6	WNW	11	6	1062	96	370	415	326	250	228
OCT	NNW	2	2	8	27	W	11	27	835	-19	234	333	263	215	174
NOV	NNW	2	3	11	11	NW	15	11	715	-67	152	269	232	181	157
DEC	NNW	2	3	15	30	N	15	30	625	-115	134	264	229	183	164
YEARLY AVERAGE	NNW	––	2	––	––	NW	15	11	––	––	––	––	––	––	212

[1] Wind speed in knots, at stated height above ground.

Annual Climatological Data for NLABS Sudbury Annex – 1963

Month	WIND[1]						SOLAR RADIATION (Langleys/Day)										
	Prevailing Dir.	Average		Maximum Hourly Average			All Wave		Vertical Eppley	Filtered 45° Eppley				Normal Incidence[2]		
	2mtr	Hourly 50cm	Speed 2mtr	Speed 50 cm	Dir. 2 mtr.	Speed 2 mtr.	Total Hem.	Net Ex.	2mtr	Clear	Yellow	Lt Red	Dk Red[1]	Clear	Yellow	Red
JAN	WNW	2	2	10	NNW	11[3]	665	-101	147	260	222	174	154	--	--	--
FEB	NW	2	3	10	NNW	13	722	-77	240	369	315	241	214	--	--	--
MAR	NW	2	3	9	WNW	11[3]	383	26	312	377	313	254	208	331	25[illegible]	186
APR	NNW	2	4	12	WNW	15	104[illegible]	151	419	456	344	282	238	419	306	229
MAY	S	2	3	7	NW	9	124[illegible]	220	502	473	370	295	257	454	347	250
JUN	NW	1	2	7	WNW	9[3]	1340	203	510	498	362	293	253	460	348	256
JUL	NW	1	1	8	NW	10	1333	219	508	508	364	305	260	399	297	233
AUG	WNW	1	2	8	WNW	10	1212	170	434	461	356	295	256	419	306	285
SEP	N	[illegible]	2	7	NW	9	1058	75	348	414	325	268	242	377	286	243
OCT	NW	2	2	11	NW	13	947	33	207	449	360	302	260	423	332	285
NOV	N	2	3	9	NW	10[3]	719	-84	115	169	141	116	99	125	96	85
DEC	N	2	3	8	NW	10	590	-140	144	266	231	189	166	249	191	166
YEARLY AVERAGE	NW	2	3	12	WNW	15	984	65	331	392	309	251	217	--	--	--

[1] Wind speed in knots at stated height above ground.

[2] Normal Incidence began in March 1963.

[3] Occurred more than once during month.

Annual Climatological Data for NLABS Sudbury Annex – 1964

Month	WIND[1] Prevailing Dir. 2mtr	Average Hourly Speed 50cm	Average Hourly Speed 2mtr	Maximum Hourly Average Speed 50cm	Maximum Hourly Average Dir. 2mtr	Maximum Hourly Average Speed 2mtr	SOLAR RADIATION (Langleys/day) All Wave Total Hem.	All Wave Net. Ex.	Vertical Eppley 2mtr	Filtered 45° Eppley Clear	Filtered 45° Eppley Yellow	Filtered 45° Eppley Lt Red	Filtered 45° Eppley Dk Red	Normal Incidence Clear	Normal Incidence Yellow	Normal Incidence Red
JAN	NW	3	3	11	WNW	13	602	123	161	301	259	209	183	--	--	--
FEB	NW	3	4	11	NW	13	697	92	253	389	318	260	229	308	244	191
MAR	NW	3	4	11	NW	12	827	27	308	386	314	254	229	309	241	171
APR	SSE	2	3	9[2]	WNW	11[2]	976	125	373	411	323	266	231	337	259	182
MAY	S	2	3	9	W	11[2]	1246	271	553	570	427	354	308	529	392	261
JUN	NW	2	2	9	NW	12	1305	300	549	492	378	310	270	528	409	262
JUL	S	1	1	5	NNW	6	1212	210	386	354	279	327	200	270	211	141
AUG	N	1	1	6	NNW	7[2]	1167	209	412	415	337	277	241	338	299	260
SEP	N	1	2	6[2]	WNW	8[2]	1083	148	361	425	345	282	246	370	289	193
OCT	NW	2	2	8	NNW	10	891	91	259	373	312	258	219	317	256	170
NOV	NW	2	2	9[2]	NW	11[2]	736	5	172	294	253	209	176	264	217	152
DEC	N	2	3	13	NW	15	641	-30	111	193	170	142	124	151	129	94

[1] Wind speed in knots at stated height above ground.

[2] Occurred more than once during month.

Annual Climatological Data for NLABS Sudbury Annex – 1965

Month	SOLAR RADIATION (Langleys/Day)									
	Total Hem.	All Wave Net. Ex.	Vertical Eppley 2mtr	Filtered 45° Eppley Clear	Yellow	Lt Red	Dk Red	Normal Incidence Clear	Yellow	Red
JAN	636	-51	155	268	230	193	169	204	175	128
FEB	743	0	239	361	312	257	227	298	247	166
MAR	840	64	295	344	297	235	214	265	217	155
APR	1007	181	410	419	351	285	246	391	304	211
MAY	1197	289	527	479	394	322	277	500	377	265
JUN	1245	287	526	451	362	361	250	466	366	252
JUL	1202	259	483	447	358	297	242	412	314	210
AUG	1158	236	423	433	341	285	236	361	268	189
SEP	987	180	302	428	339	266	237	358	279	174
OCT	820	60	188	403	327	272	238	354	280	194
NOV	664	-13	163	282	233	198	173	260	204	144
DEC	654	10	126	227	191	161	142	190	154	114
YEARLY AVERAGE	929	123	320	379	311	256	221	338	266	184

Annual Climatological Data for NLABS Sudbury Annex – 1966

	WIND[1]				SOLAR RADIATION (Langleys/Day)									
					All Wave			Filtered 45° Eppley				Normal Incidence		
Month	Prevailing Direction	Average Hourly Speed	Max. Hourly Speed	Conc Dir	Total Hemph	Net Exch	Vertical Eppley	Clear	Yellow	Lt Red	Dk Red	Clear	Yellow	Red
JAN	WNW	2	8[2]	NNW	610	-44	165	287	234	200	174	243	185	133
FEB	NNW	2	9	NNW	692	9	227	337	269	229	197	260	203	141
MAR	N	2	9	WNW	867	136	319	407	318	270	230	344	268	179
APR	NNW	2	7[2]	WNW	936	190	369	386	300	247	208	271	211	145
MAY	SSE	2	9	NW	1081	249	463	427	327	270	225	361	276	187
JUN	N	1	6[2]	NNW	1235	335	493	445	340	276	237	402	312	204
JUL	SSW	1	5[2]	WNW	1299	349	548	512	404	321	272	496	384	254
AUG	WNW	1	6[2]	NNE	1172	259	444	459	361	292	240	413	312	212
SEP	N	1	8	SSE	1024	167	350	417	339	271	230	370	283	185
OCT	NW	1	10[2]	WNW	841	56	257	385	318	262	224	353	267	180
NOV	NNW	2	10[2]	NNW	718	-28	158	266	225	185	163	230	181	127
DEC	NNW	2	12	SE	617	-37	136	260	220	186	162	225	176	125

[1] Wind speed in knots.

[2] Occurred more than once during month.

Annual Climatological Data for NLABS Sudbury Annex – 1967[1]

Month	WIND[2]				SOLAR RADIATION (Langleys/day)										
		Average Max			All Wave			Filtered 45° Eppley				Normal Incidence		
	Prev Dir	Hourly Speed	Hourly Speed	Conc Dir	Total Hemph	Net Exch	Vertical Eppley	Clear	Yellow	Lt Red	Dk Red	Clear	Yellow	Red
JAN	SW	1	6[3]	WNW	641	-36	136	232	184	154	129	178	138	98
FEB	WNW	3	13	W	644	-25	226	330	263	218	187	271	206	148
APR	NNW	4	13[3]	NNE	918	191	339	411	342	289	251	383	281	194
JUN	SW	1	6[3]	N	1134	291	489	408	333	277	251	332	255	174
JUL	SSW	1	5	NNW	1157	253	468	411	326	276	241	231	184	129
AUG	SSW	1	7	N	1077	202	422	363	309	266	234	242	198	135
SEP	NNW	1	8[3]	NNW	996	152	396	463	367	325	287	412	317	214
OCT	SSE	1	7[3]	SSE	853	77	269	356	308	264	228	300	228	161
NOV	W	2	8[3]	NW	692	-21	161	260	231	198	171	193	151	110
DEC	NW	2	10[3]	N	643	-30	136	243	220	189	166	227	178	131

[1] Data for March and May are missing.

[2] Wind speed in knots.

[3] Occurred more than once during the month.